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IS 13574 (1992): Animal Feeds and Feeding Stuffs -
Determination of Calcium and Magnesium in Mineral
Supplements [FAD 5: Livestock Feeds, Equipment and Systems]



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भारतीय मानक

पशु आहार तथा भरण सामग्री — खनिज अनुपूरकों में
कैल्शियम तथा मैगनीशियम ज्ञात करना

Indian Standard

ANIMAL FEEDS AND FEEDING STUFFS —
DETERMINATION OF CALCIUM AND
MAGNESIUM IN MINERAL SUPPLEMENTS

UDC 636.085.12 : 543 [546.41 + 546.46]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Livestock Feeds Sectional Committee had been approved by the Food and Agriculture Division Council.

The test method for determination of calcium as prescribed in 5 of IS 7874 (Part 2) : 1975 has been revised since during usage of this test method, it was reported to give low recovery.

IS 13433 : 1992 'Animal feeds and feeding stuffs — Determination of calcium' replaces the contents given under 5 of IS 7874 (Part 2) : 1975 and has been published in two parts:

Part 1 Titrimetric method; and

Part 2 Atomic absorption spectrometric method.

This Indian Standard which is applicable to mineral supplements only, prescribes the EDTA method for determination of calcium and magnesium.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ANIMAL FEEDS AND FEEDING STUFFS — DETERMINATION OF CALCIUM AND MAGNESIUM IN MINERAL SUPPLEMENTS

1 SCOPE

This Indian Standard prescribes the EDTA method for the determination of calcium and magnesium in mineral supplements.

Due to lower concentration of calcium in animal feeds and fodders and because of high organic matter content, the procedure is not suitable for feeds and fodders.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this Standard:

<i>IS No.</i>	<i>Title</i>
460 (Part 1) : 1985	Specification for test sieves : Part 1 Wire cloth test sieve
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)

3 PRINCIPLE

Titration of the test sample with EDTA using ammonium purpureate as an indicator is done to determine the calcium content of the sample. Titration of another test sample with EDTA using Eriochrome Black T as indicator is done to determine the total calcium and magnesium content. EDTA reacts first with free calcium ions and finally with magnesium indicator complex, changing the wine red colour to blue. Magnesium content is calculated as the difference of the two determinations.

4 REAGENTS

4.1 Magnesium Chloride Hexahydrate — AR grade.

4.2 Ethylene-diamine-tetra-acetate (Versenate), EDTA Solution 0.01 N

Dissolve 2.0 g of disodium dihydrogen ethylene-diamine-tetra-acetate and 0.05 g of magnesium chloride hexahydrate in water and dilute to a volume of 1 litre. Standardize the solution against calcium chloride using Eriochrome T indicator for using it for calcium and magnesium estimation and with ammonium purpureate for using it for calcium estimation.

4.3 Ammonia Concentrate

Specific gravity 0.88 — 0.90.

4.4 Ammonium Chloride : Ammonium Hydroxide Buffer

Add 142 ml of concentrate ammonia (sp. gr. 0.88-0.90) to 17.5 g of ammonium chloride (AR grade) and dilute the volume after solubilization to 250 ml in a volumetric flask using distilled water. This solution should have an approximate pH of 10 which can be verified with the help of a pH paper.

4.5 Hydrochloric Acid (25 Percent)

Take 60 ml of concentrated hydrochloric acid in a 100 ml cylinder and make the volume up to the mark with distilled water.

4.6 Sodium Hydroxide (4 N)

Dissolve 160 g of sodium hydroxide in 1 litre of distilled water.

4.7 Ammonium Purpureate Indicator

Thoroughly mix 0.5 g of ammonium purpureate (AR grade) with 100 g of powdered potassium sulphate (AR grade).

4.8 Eriochrome Black T-Indicator

Dissolve 0.2 g of dye in 15 ml of triethanolamine and add 5 ml of absolute alcohol.

4.9 Standard Calcium Chloride Solution 0.01 N

Dissolve 0.5 g of pure calcium carbonate in 10 ml of approximately 3 N hydrochloric acid and dilute to a volume of one litre.

4.10 Nitric Acid — concentrated.

5 PROCEDURE

5.1 Sample Preparation

Weigh 1.0 g of the sample to be analyzed in the beaker. Add 40 ml of 25 percent HCl and a drop or two of the concentrated nitric acid. Boil for a few minutes and allow to cool. Filter through Whatman filter paper No. 41 in 250-ml volumetric flask. Give final wash to the filter

paper with distilled water and collect the washings in the volumetric flask. Make up the volume with distilled water and mix thoroughly. Take 25 ml of the solution in another 100-ml flask and make the volume with distilled water.

5.2 Determination of Calcium

Pipette an aliquot of 25 ml of the test sample (5.1) into a conical flask. Dilute it with 50 ml of water. Add about 2.0 ml or 20 drops of 4N sodium hydroxide solution and 50 mg of ammonium purpureate indicator. Titrate with 0.01 N EDTA till the colour changes from wine red to purple.

5.3 Determination of Total Calcium and Magnesium

Pipette an aliquot of 25 ml of the above test sample (5.1) and pour into a 250-ml conical flask, dilute to about 50 ml with distilled water. To this add 0.5 ml or 10 drops of buffer solution. Add 3-4 drops of Eriochrome T-indicator and titrate with 0.01 N EDTA until the colour is changed from wine red to clear blue. Titration is carried out slowly at the end point.

6 CALCULATIONS

6.1 Calcium

$$\text{Milliequivalent of calcium per litre (A)} = \frac{X \times N \times 1\,000}{V}$$

where

X = volume of EDTA solution used for titration (see 5.2),

N = normality of EDTA (0.01 N), and

V = volume of aliquot of test sample.

6.2 Calcium and Magnesium

$$\text{Milliequivalent of total calcium and magnesium per litre (B)} = \frac{X \times N \times 1\,000}{V}$$

where

X = volume of EDTA solution used for titration (see 5.3),

N = normality of EDTA (0.01 N), and

V = volume of aliquot of test sample.

6.3 Magnesium

$$\text{Milliequivalent of magnesium (C)} = B - A$$

where

B = milliequivalent of total calcium and magnesium per litre (see 6.2); and

A = milliequivalent of calcium per litre (see 6.1).

7 EXPRESSION OF RESULTS

$$\text{7.1 Calcium, percent} = A \times 20 \times 100$$

where

A = milliequivalent of calcium per litre (see 6.1), and

20 = equivalent weight of calcium.

$$\text{7.2 Magnesium, percent} = C \times 12 \times 100$$

where

C = milliequivalent of magnesium per litre (see 6.3), and

12 = equivalent weight of magnesium.

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